

**Analysis of Total Suspended
Particles (TSP) and Total Organic
Carbon (TOC) in Air Samples:**

**Integrated Atmospheric
Deposition Network (IADN)
TSP/TOC Procedure**

**Michael Wassouf and Ilora Basu
School of Public and Environmental Affairs
Indiana University
Bloomington, IN 47405**

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1.0 Introduction

Air particulates are collected from three sites: Eagle Harbor, 100 meters from Lake Superior, Michigan on the Keweenaw Peninsula; Sleeping Bear Dunes, on Lake Michigan 5 km south of Empire, Michigan; and Sturgeon Point, 25 km southwest of Buffalo, New York and 100 meters from Lake Erie. Air is drawn through the Whatman quartz microfibre filter, 20.3 x 25.4 cm at a flow rate of 68 m³/hr for 24 hours using an Anderson Hi-Vol air sampler. All particles greater than two microns are retained by the filter. The filters are then transported to Indiana University where they are analyzed for the total suspended particle and total organic carbon.

2.0 Supplies and Equipment:

2.1 Supplies

2.1.1 Quartz Microfibre Filters (Whatman 20.3 x 25.4 cm)

2.1.2 LiNO₃

2.1.3 Aluminum foil

2.1.4 Tweezers

2.1.5 Hexane (EM Science, Omnisolv)

2.1.6 Plastic Zip-Lock bags

2.2 Equipment

2.2.1 Balance (Mettler AE 50)

2.2.2 Mettler Balance GD Hanger

2.2.3 Humidity Chamber (Lab-Line Desicab No. 1477) with wet LiNO₃ in a tray.

2.2.4 Leco Total Carbon Analyzer

2.2.5 Muffle Furnace (Thermolyne Type 30400)

3.0 Balance Calibration

- 3.1 The balance must be connected to the power supply for at least 60 minutes before calibrating.
- 3.2 Press and hold the single control bar until -CAL- appears in the display, then release the control bar. The display changes to CAL----, followed by CAL 50 (blinks).
- 3.3 Move calibration lever all the way to the rear; the display changes to CAL----, followed by 50.0000, then to CAL 0 (blinks).
- 3.4 Move calibration lever all the way back to the front of the balance; the display changes to ----, followed by 0.0000.

4.0 Filter Preparation Before Sampling

- 1.1 Wrap filters in aluminum foil (shiny side out).
- 4.2 Muffle filters for four to six hours @ 450°C, store in freezer until use.
- 4.3 Open foil slightly and place filters in desiccator (50% humidity via LiNO₃) for 24 hours.
- 4.4 Re-zero balance.
- 4.5 Write *Filter ID#* in top right corner of filter with pencil.
- 4.6 Open balance hanger and insert filter (unwrapped) using tweezers rinsed in hexane; close door.
- 4.7 Wait until balance equilibrates and record mass in *Filter Log Book* as *Initial Weight*.
- 4.8 Remove filter from balance and re-zero.
- 4.9 Weigh filter again; if mass is within 0.1 mg of first mass go on, if not repeat weighing until two measurements within 0.1 g are taken and record the average as *Initial Weight*.
- 4.10 Re-wrap filter in original piece of aluminum foil .
- 4.11 Write *Filter ID#* on foil with magic maker.
- 4.12 Place in plastic zip-lock bag.
- 4.13 Repeat 1-12 for each filter.

5.0 TSP Measurement on Filter

- 5.1 Remove filter from plastic bag; open foil slightly and place in desiccator for 24 hours.
- 5.2 Record *Sample ID Code* with corresponding *Filter ID#* in *Filter Log Book* and on plastic bag.
- 5.3 Weight filter as above.
- 5.4 Record mass as *Final Weight*
- 5.5 Calculate and record *TSP (Final Weight - Initial Weight)*.
- 5.6 Re-wrap filter in original foil and plastic bag.
- 5.7 Place in cold room or freezer until TOC analysis.

6.0 Preparing Filters for TOC Analysis

- 6.1 Remove filter from bag and foil.
- 6.2 Cut six discs, 1.9 cm diameter, from filter with cork borer and place discs in a plastic petri dish.
- 6.3 Record *Sample ID Code* on petri dish.
- 6.4 Record *TSP* from *Filter Log Book* in *TSP/TOC Log* as *TSP on Filter*.
- 6.5 Calculate and record *TSP on Circle* ($TSP\ on\ Filter \times 2.84/404$). The area of the whole filter is 404 sq. cm. The area of the circle is 2.84 sq. cm.
- 6.6 Multiply *TOC* by six, this is the number you will enter into the Leko Carbon Analyzer.

7.0 TOC Analysis: Using LEKO Total Carbon Analyzer

See following pages (from Leco operator's manual).

CALIBRATION

Leco needs calibration for both the balance and the standards. This is performed at the beginning of each day that Leco will be used.

To calibrate the balance:

1. Press the "system update" key.
2. Press the "5" key.
3. Place a crucible on the balance.
4. Press the "tare" key.
5. Place a 1 gram weight in the crucible; the readout will flash.
6. Remove the 1 gram weight.

Leco needs calibration with carbon and sulfur standards before combusting samples. To calibrate for carbon and sulfur, the standards will be combusted using the same procedure used for samples (see Sample Combustion). A minimum of 5 standard combustions are used for the calibration. The values for these combustions should be within 0.05g of each other. When 5 consistent values have been obtained, perform the following procedure for calibration:

1. Press the "system update" key. Message center will display "display contents?". Press the "1" key.
2. "Auto Calibrate By Stack" will be displayed on message center. Press the "Yes" key.
3. "Carbon recalibrate" will be displayed on the message center. If you are calibrating for carbon, press the "Yes" key. If you are calibrating for sulfur, press the "No" key. The message center will now display "Sulfur Recalibrate." Press the "Yes" key.
4. Message center will now display "Calibrate By Standard." Press the "Yes" key. Message center will ask the value of the standard. Press the "Enter" key.
5. A complete answer stack for the last 10 analyses will be printed.
6. The print out list will be displayed on the message center one value at a time. Press the "Yes" key for values to be included, and the "No" key for values to be excluded.
7. A revised answer list will print out at the end of calculations. Check this list against the original answer list to be sure Leco has recalibrated.

SAMPLE COMBUSTION, STANDARD PROCEDURE

Leco must warm up for one hour before use. Both the furnace and measurement units need power switches turned on. Be sure oxygen and compressed air valves are open before beginning combustions.

To combust samples, you will need:

1. Crucibles
2. Lecocel
3. Iron Chip Accelerator

To combust samples, perform the following:

1. Place a crucible on the balance. Do not touch the crucible with fingers.
2. Measure 250 mg of the sample into the crucible. Mass will be displayed on message center.
3. Press the “Enter” key. Mass will be moved to the left side of the display.
4. Using tongs, remove the crucible from the balance. Tap to distribute sample evenly on the bottom of the crucible.

SAMPLE COMBUSTION, MANUAL WEIGHT PROCEDURE

Leco must warm up for one hour before use. Both the furnace and measurement units need power switches turned on. Be sure oxygen and compressed air valves are open before beginning combustions.

To combust samples, you will need:

1. Lecocel
2. Iron Chip Accelerator

To combust samples, perform the following:

1. Press the “Manual Weight” key.
2. Enter the sample weight using the keyboard.
3. Press the “Enter” key. Mass will be moved to the left side of the display.
4. Using tongs, remove the crucible from the balance. Tap to distribute sample evenly on the bottom of the crucible.

